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PATENT SPECIFICATION

NO DRAWINGS

1051514

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COMPLETE SPECIFICATION

Process for preventing the Adhesion of Water Soluble Films

We, DENKI KAGAKU KOGYO KABUSHIKI KAISHA, of No. 10, 1-Chome, Yuraku-Cho, Chiyoda-Ku, Tokyo, Japan, a company organized according to the laws of Japan, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

THIS INVENTION relates to a process for preventing the adhesion of water soluble polyvinyl alcohol film which film is made by melt extrusion.

It has been known that polyvinyl alcohol (abridged as PVA hereinafter) has been utilized as a water soluble film by the so-called casting process, wherein an aqueous solution of PVA is cast and then dried, this is because the PVA in which there are acetic acid radicals left to a certain extent dissolve easily in cold water.

The inventors have found that a water soluble PVA film can be manufactured economically and advantageously by the melt extrusion process under anhydrous condition by using a partially saponified PVA. According to the melt extrusion process, not only sheet film but also cylindrical film can be manufactured as PVA film.

However, it has been observed that PVA films manufactured by the melt extrusion process absorb moisture in air and adhere to each other at a high humidity in a similar way to PVA films made by the casting process.

For the previous film which is manufactured by the casting process, there is a known process in which the surface of sheet is subjected to the aventurine processing or a process in which aliphatic dicarboxylic acids being to a certain extent water soluble are added to an aqueous solution of PVA. Such a process is not always effective in the manufacture of water soluble PVA films by the melt extrusion method. This is because in the inner surface of cylindrical film which is

manufactured by the melt extrusion method, it is difficult to attain an effective prevention of adhesion by the aventurine processing and the addition of said dicarboxylic acid does not show a great effect as shown in the control example described hereinafter.

As a result of various investigations with regard to a process for preventing the adhesion of water soluble films made by the melt extrusion process it has been found that the adhesion of film can be prevented without decreasing the solubility of film in water by melt extending a composition comprising polyvinyl alcohol and an agent consisting of at least one of a monobasic higher fatty acid containing more than 10 carbon atoms, a salt, ester, amide, or methylol amide thereof, or a higher aliphatic alcohol containing more than 10 carbon atoms.

More particularly, the invention consists of mixing one or more of monobasic higher fatty acid containing more than 10 carbon atoms, a salt, ester, amide, or methylol amide thereof, or a higher aliphatic alcohol containing more than 10 carbon atoms with powders of PVA by sufficient dispersion and melt extending the resulting composition. It is not necessary to add these agents in a great amount and in general a mixing ratio below 1% shows a sufficient effect of preventing the adhesion. The use of said agent in a great amount makes the film turbid and shows rather less effect of preventing the adhesion as expected.

When said composition in which such agents are thoroughly dispersed is melt extruded, a film showing good effect of preventing adhesion may be obtained.

According to the invention, a sufficient degree of prevention of adhesion can be attained with an amount of said agents and the interior adhesion of cylindrical film can be prevented. Even if the present film is left for a long time, the effect is not reduced and the additives do not separate on the sur-

face of film to form turbidness.

EXAMPLE

- 5 A mixed liquid of 9 parts of vinyl acetate, 1 part of ethylene glycol, 2 parts of methanol and 0.005 part of azobisisobutyronitrile was polymerized at a polymerization temperature of 65°C for 8 hours and the vinyl acetate monomer was distilled off by a conventional method, after the polymerization.
- 10 A methanolic solution of polymer of 45% concentration was then prepared and saponified with caustic soda of 1/220 equivalent per mol of vinyl acetate at a temperature of 25°C to yield PVA having a polymerization degree of 900 and residual acetic acid radical of 16 mol %.

15 To the resulting PVA was added a solution of each agent shown in the following table

in methanol or in a mixture of methanol and acetone, and dried in a vacuum drier at 60°C and under a reduced pressure of 10 mmHg after mixed thoroughly.

The dried PVA was thrown into a melt extruder of 15 mm screw diameter and 300 mm length ($L/D = 20$) and moulded at 200°C. at a rate of 200 g per hour from a ring die of 25 mm diameter and 0.3 mm clearance to yield a colourless, transparent and water soluble film.

Two sheets of said film were plied under a load of 95 g/cm², left for 20 hours in a thermo-hygrostat at 20°C and 80% RH and then the strength for stripping the films was determined to observe the effect of preventing the adhesion. The results are shown in the following table.

TABLE

No.	Additive	Amount added (wt. %) based on wt. of PVA	Strength of stripping film (g/cm)
1	No	—	54.8
2	Lauric acid	1.0	7.3
3	Stearic acid	1.0	3.1
4	" "	0.75	3.2
5	" "	0.5	9.3
6	Linoleic acid	1.0	11.7
7	Sodium stearate	0.5	8.7
8	Stearyl Alcohol	0.2	13.6
9	Stearic acid amide	1.0	2.3
10	Stearic acid methylol amide	1.0	3.5
11	Lauryl stearate	0.2	4.3
12	Stearic acid + stearic acid amine	0.5 + 0.5	2.3
Control 1	Caprylic acid	1.0	44.3
" 2	Sebacic acid	1.0	36.9

- 40 It is obvious from the results that the addition of one or more of monobasic higher fatty acid having more than 10 carbon atoms, salt, ester, amide, or methylol amide thereof, or a higher alcohol having more than 10 carbon atoms shows sufficient effects on the prevention of adhesion of water soluble PVA films made by the melt extrusion process.

WHAT WE CLAIM IS:—

1. A process for preparing a water soluble polyvinyl alcohol film by melt extrusion which process comprises melt extruding a composition comprising polyvinyl alcohol and an agent consisting of at least one of a monobasic higher fatty acid containing more than 10 carbon atoms or a salt, ester, amide or

methylol amide thereof or a higher aliphatic alcohol containing more than 10 carbon atoms.

2. A process according to claim 1 in which the nature and proportion of said agent is such that self adhesion of the polyvinyl alcohol film produced is prevented.
- 5 3. A process according to claim 1 or 2 in which the composition is prepared by mixing the said agent with the polyvinyl alcohol in powder form.
- 10 4. A process according to any one of claims 1-3 in which the said agent is present in an amount below 1%.
- 15 5. A process according to any of claims 1-4 wherein said agent is lauric acid.
6. A process according to any of claims 1-4 wherein said agent is stearic acid.
7. A process according to any of claims 1-4 wherein said agent is linoleic acid.
- 20 8. A process according to any of claims 1-4 wherein said agent is sodium stearate.
9. A process according to any of claims 1-4 wherein said agent is stearyl alcohol.
- 25 10. A process according to any of claims 1-4 wherein said agent is stearic acid amide.

11. A process according to any of claims 1-4 wherein said agent is stearic acid methylol amide.

12. A process according to any of claims 1-4 wherein said agent is lauryl stearate.

13. A process of preparing polyvinyl alcohol films as claimed in claim 1 and substantially as hereinbefore described in the example.

14. A non adhesive water soluble film of a polyvinyl alcohol prepared by melt extrusion of composition comprising polyvinyl alcohol and at least one of a monobasic higher fatty acid containing more than 10 carbon atoms, or a salt, ester, amide or methylol amide thereof or a higher aliphatic alcohol containing more than 10 carbon atoms.

15. Polyvinyl alcohol films when prepared by the process claimed in any one of claims 1 to 13.

MARKS & CLERK,
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